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ORIGINAL ARTICLE

Effects of three luting agents and cyclic impact loading on shear bond strengths to zirconia with tribochemical treatment

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bond strength;
cyclic impact loading;
resin cement;
tribochemical treatment;
zirconia

Abstract *Background/purpose:* The purpose of this study was to investigate the effects of cyclic impact loading on shear bond strengths of three different luting cements to zirconia.
Materials and methods: The following cements were used in this study for bonding two zirconia blocks that were tribochemically silica-coated with the Rocatec system: Super Bond C&B (SB); Panavia Fluoro Cement (PF); and Fuji Luting (FL). Specimens were subsequently subjected to three storage conditions: 10^6 compressive cyclic impact loading (CL), 10^6 shear cyclic impact loading (SL), and no cyclic impact loading (Control) in distilled water at 37 °C with a mechanical fatigue testing device. Shear bond strength tests were performed with a universal testing machine.
Results: Bond strengths of PF+Control (63.6 $\pm$ 2.4 MPa) and PF+CL (57.2 $\pm$ 3.8 MPa) were significantly higher than those with SB and FL. There was no significant difference between CL and SL ($P > 0.05$) with SB and FL. The bond strength of the FL resin-modified glass ionomer cement was significantly lower than those with PF and SB. After applying tribochemical treatment, all specimens subjected to cyclic impact loading exhibited cohesive failure in the resin cement.
Conclusions: After 10^6 compressive cyclic impact loading, the highest bond to zirconia was obtained with 10-methacryloxydecyl-dihydrogenphosphate containing a luting system (Panavia

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Fluoro Cement). After applying tribochemical treatment to zirconia, all cements used in this study survived more than 10^6 shear or compressive cyclic impact loading at 10 kg.

Introduction

Interest in using high-strength zirconium oxide ceramics for oral rehabilitation has grown in recent years.[1,2] Zirconia has the most favorable properties with a flexural strength of 900 MPa to1200 MPa, a fracture resistance of >2000 N, and a fracture toughness of 9 to 10 MPa[$m^{0.5}$/ok?], which is almost twice the value obtained for alumina-based materials.[3] Computer-assisted design and manufacture technologies make working with this high crystalline material simpler, allowing the fabrication of full-coverage crowns and bridge frameworks.[4]

The problem related to the performance of high-strength zirconia is that adhesion of resin cements to such ceramics is [debatable/uncertain?]. In several studies on zirconia-ceramic bonding, air-abrasion was used to condition the ceramic surface in order to increase the surface roughness, and to clean and activate the surface.[5,6] This method can significantly improve resin-zirconia ceramic bond strength and durability by increasing surface roughness, and cleaning and activating the ceramic surface when combined with adhesive monomer-containing primers such as 10-methacryloxydecyl-dihydrogenphosphate (MDP).[6] Lin et al[7] evaluated tribochemical treatment (also known as Rocatec treatment) and proved that it is an effective surface pretreatment method that improves the bond strength of zirconia. Tribochemical treatment conditions the surface by fusing a unique silica layer onto the surface of a coping or restoration, creating a silica-coated surface for enhanced bond strength.

Kern et al[8] evaluated different methods and materials for bonding resin composite luting cements to zirconia, while others tested the durability of bonding after long-term water storage and thermal cycling.[7–12] However, dental restorations are clinically subjected to cyclic forces ranging from 60 N to 250 N during function and as high as 500 N to 800 N for short periods.[13] Distributions of stresses generated around a bonding interface may have great influences on fracture patterns. Therefore, it is critical to investigate bond strength values obtained under static conditions and values obtained under dynamic cyclic impact loading caused by occlusal masticatory forces.

Unfortunately, previous research failed to consider the effects of cyclic impact loading.[14–16] *In vitro* simulated impact cyclic load tests[15,16] are less time consuming and costly than *in vivo* studies. A primary goal is standardization of *in vitro* specimens so that results of studies can be compared. The use of extracted natural teeth as specimens more closely simulates clinical conditions.[17] However, standardization of natural teeth is difficult, while use of Y-TZP ceramic blocks is easier to standardize.[15] The technique of using a computer-controlled masticatory simulator and exposing ceramic material to 1.2×10^6 cycles to simulate a 5-year in vivo period has been demonstrated successfully in several investigations.[18,19] The current paper focuses on cyclic impact loading and evaluated shear bond strengths of three different luting cements to zirconia.

Materials and methods

Specimen preparation

Two different sized ($10 \times 10 \times 20$ mm and $10 \times 10 \times 10$ mm) quadrate yttrium oxide-stabilized polycrystalline tetragonal zirconia ceramic (Y-TZP) blocks (5.03 wt% Y_2O_3 and 94.67 wt% ZrO_2; Nikkato, Tokyo, Japan) were prepared ($n = 81$). The zirconia surfaces were flattened and polished through the following grit sequence: 220 SiC, 400 SiC, and 600 SiC. Specimens were distributed in nine different test groups according to the cement type and impact loading conditions ($n = 9$ per group).

The following luting cements for bonding zirconia (Table 1) were used in this study: a 4-methacryloxyethyl trimellitic acid adhesive resin, Super Bond C&B (SB; Sun Medical, Tokyo, Japan); a resin composite containing phosphoric acid monomers, Panavia Fluoro Cement (PF; Kuraray Medical, Osaka, Japan); and a resin-reinforced glass ionomer cement, Fuji Luting (FL; GC, Tokyo, Japan).

The impact loading conditions were as follows. In the Control group, a shear bond strength test was performed after storage in distilled water at 37 °C for 12 days prior to bond tests. In the CL group, a shear bond strength test was performed after compressive cyclic impact loading of 10^6 cycles at 1 Hz between 0 and 98 N (for approximately 12 days). The load was applied parallel to and at the central part of the bonding surface in distilled water at 37 °C. In the SL group, the shear bond strength test was performed after shear cyclic impact loading of 10^6 cycles at 1 Hz between 0 and 98 N (for approximately 12 days). The load was applied perpendicular to and at the edge part of the bonding surface in distilled water at 37 °C.

The polished (600 grit SiC) surfaces of zirconia blocks were conditioned with: (1) tribochemical silica coating with Rocatec-Plus for 13 s at 0.25 MPa; (2) air cleaning for 5 s; and (3) silane coating with Espe-SIL (3M ESPE, St Paul, MN, USA) with a brush. They were then allowed to dry in ambient air for 5 min before cement was applied to the specimens.

Bonding procedure

After appropriate surface treatment, each adhesive resin cement was applied according to the manufacturers' instructions at room temperature (23.0 ± 1.0 °C) and

Table 1 List of materials used in this study.

Product/Manufacturer/Lot number	Code	Main composition
Resin cements		
Super Bond C&B/Sun Medical/ Powder: TR1, Liquid: TR1, Catalyst: TM42	SB	Powder: PMMA, Liquid: MMA, 4-META, Catalyst: TBB
Panavia Fluoro Cement/Kuraray Medical/ Paste A: 00209A, Paste B: 00116A	PF	Paste A: BPEDMA, MDP, DMA, silica, barium, sulfate, dibenzoylperoxide, Paste B: N,N-diethanol-p-toluidine, silica sodium fluoride
Oxyguard II/Kuraray Medical/ 00447A		Polyethyleneglycol, glycerine, sodium, benzenesulfinate cont. gel
Resin-reinforced glass ionomer cement		
Fuji Luting/GC/212182	FL	Alumino-silicate, polyacrylic acid, HEMA
Rocatec System		
Rocatec-Plus/3M ESPE/142820		110 μm silica containing alumina particles
Espe-SIL/3M ESPE/148638		Silane

BPEDMA = bisphenol A polyethoxy dimethacrylate; HEMA = 2-hydroxyethyl methacrylate; MDP = 10-methacryloyloxy-decyl dihydrogenphosphate; 4-META = 4-methacryloxyethyl trimellitic acid; MMA = methyl methacrylate; PMMA = polymethyl methacrylate; TBB = tri-n-butylborane.

a relative humidity of 50% ± 5%. Bonding procedures followed the manufacturers' recommendations. Two zirconia blocks were bonded to each other under a load of 147 N (15 kg) for 15 min in order to standardize the applied pressure. Excess resin cement was removed with a laboratory knife. The bonded area was 100 mm^2.

A customized fatigue tester was constructed for this study; Fig. 1 shows the fatigue tester, the supporting metallic jig, and a schematic diagram. The maximum applied load for any test load could be varied from 3 kg to 50 kg. For this experiment, the cycle frequency was fixed at 75 cycles/min, the vertical movement was 6 mm, and the descending speed 30 mm/s.[20] The test station had an independent counter that was automatically halted when a specimen fractured. One-third of the specimens were subjected to 10^6 compressive cyclic impact loading (Fig 1B) and shear cyclic impact loading (Fig. 1C). Specimens were kept in water at 37 °C and subjected to shear cyclic loading test regimes of 10 kg using the fatigue-testing machine. Cycling continued until failure, and the time required for specimen failure was recorded. When a sample survived, the test was halted after 10^6 cycles. The control group was stored in distilled water at 37 °C for 12 days prior to the bond tests.

Shear bond strength test

Specimens were subjected to the shear bond strength test at a crosshead speed of 0.5 mm/min until fracture occurred. Fig. 2 shows the load cell for the shear bond strength test used in this study. Shear bond strength was determined according to ISO/TS 11405:2003 using a Universal Testing Machine (Servopulser EHF-FD1; Shimadzu, Kyoto, Japan). The force at separation (N) was divided by the cross-sectional area (100 mm^2) to provide results in units of stress (MPa). After debonding, the fractured interfaces of the specimens were examined with a light microscope (Leica MZ7.5; Leica Microsystems, Wetzlar, Germany) at 40× magnification to determine the debonding mode of either adhesive failure at the zirconia surface or cohesive failure in the resin cement.

Statistical analyses

Two-way analysis of variance (ANOVA) and Tukey's honest significant difference test were used to analyze the data (SPSS for Windows version 10.0; SPSS, Chicago, IL, USA) with the shear bond strength as the dependent variable.

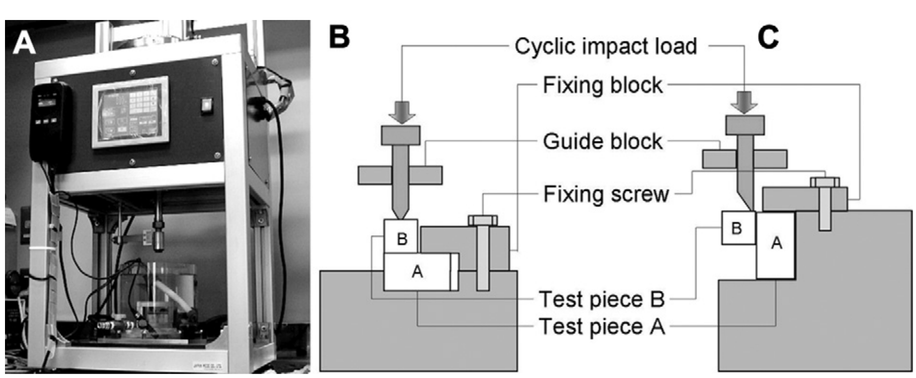

Figure 1 (A) Cyclic impact testing machine; (B) load cell of compressive cyclic impact load; (C) shear cyclic impact load.

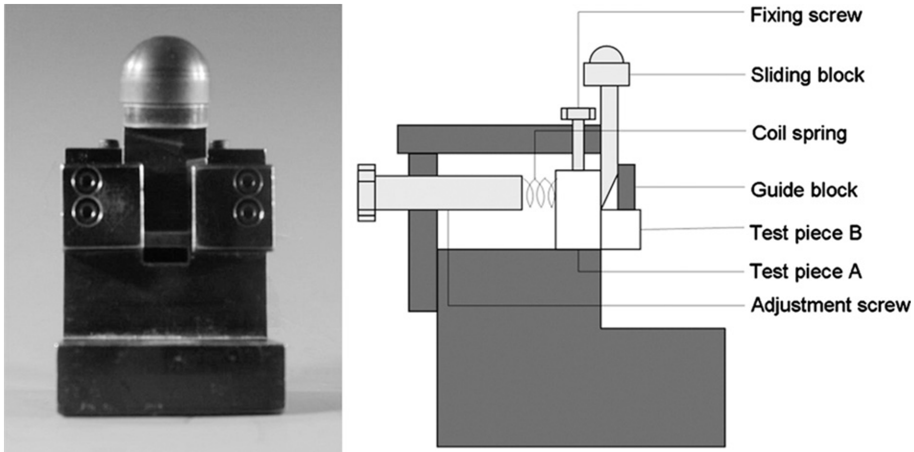

Figure 2 Load cell of shear bond strength test used in this study. Fixture for holding the specimen and aligning the test interface parallel to the sliding block for shear bond strength test.

P values of $<$0.05 were considered statistically significant in all tests.

Scanning electron microscope examination of debonded specimens

Zirconia surfaces polished with 600-grit polishing paper, modified zirconia surfaces after tribochemical treatment, and representative fractured surfaces were observed under a scanning electron microscopy (SEM) (S-4000; Hitachi, Tokyo, Japan) with an acceleration voltage of 5 kV after sputter coating using a gold-palladium alloy conductive layer (Ion sputter E-1030; Hitachi, Tokyo, Japan).

Results

Bond strength results were significantly affected by the impact load condition and type of luting agent ($P < 0.01$; ANOVA). Table 2 shows the shear bond strengths for the nine groups. The bond strengths of PF+Control and PF+CL were significantly higher than those with SB and FL. For SB and FL, bond strengths for control conditions (SB+Control and FL+Control) were significantly ($P < 0.05$) higher than those for the SL and CL groups (SB+SL, SB+CL, FL+SL, and FL+CL). However, there was no significant difference between CL and SL ($P > 0.05$) with SB and FL. Bond strengths of resin-modified glass ionomer FL cements were significantly lower than those with PF and SB.

No specimens failed during cycling. A micrograph of the modified zirconia surface after tribochemical treatment showed abundant particles and microporosities (Fig. 3B). Representative SEM images of fractured surfaces after the shear bond strength test are shown in Fig. 4. All specimens subjected to cyclic impact loading exhibited cohesive failure in the resin cement. Under CL and SL conditions, SB showed the growth of dimples, which were deeper and larger than those in the control conditions. PF and FL showed irregular cracks. FL+SL exhibited the most cracks.

Table 2 Groups and means ± standard deviations of shear bond strength.

Groups	Shear bond strength (MPa)
SB+Control	53.8 ± 2.5 a
SB+CL	46.3 ± 2.4 d
SB+SL	45.5 ± 8.7 d
PF+Control	63.6 ± 2.4 c
PF+CL	57.2 ± 3.8 b
PF+SL	45.2 ± 2.1 d
FL+Control	35.9 ± 1.7 e
FL+CL	32.4 ± 1.9 f
FL+SL	31.3 ± 4.0 f

Means identified by the same lower case letters indicate no significant difference ($P > 0.05$).

Discussion

Long-term water storage at a constant temperature[21] or thermal cycling[8] are the most often used conditions to simulate aging of resin bonds. However, the effects of occlusal forces cannot be neglected. Mean masticatory forces were reported by Anderson[22] to be in the range of 70.6 N to 146.1 N. The frequency of the loading device conformed to the masticatory rate of 60 to 120 strokes/min reported by Graf.[23] In this study, a dynamic load of 10^6 cycles at 1 Hz from 0 N to 98 N was used to test the durability of three luting cements.

When an adhesive-adherent specimen is subjected to testing by shear or tensile forces, fracture is expected to occur at the interfacial zone, and the bond strength is thereby determined. Shear bond strength tests were criticized for developing nonhomogeneous stress distributions at the bonding interface.[24] With shear testing, failure often begins in one of the substrates and not at the adhesive zone, inducing either an underestimation or a misinterpretation of the results. However, these conclusions were drawn from previous studies in which bond-strength tests were conducted on dentin or glass-ceramic substrates.[25–27] Results from tensile tests are reported to be greatly

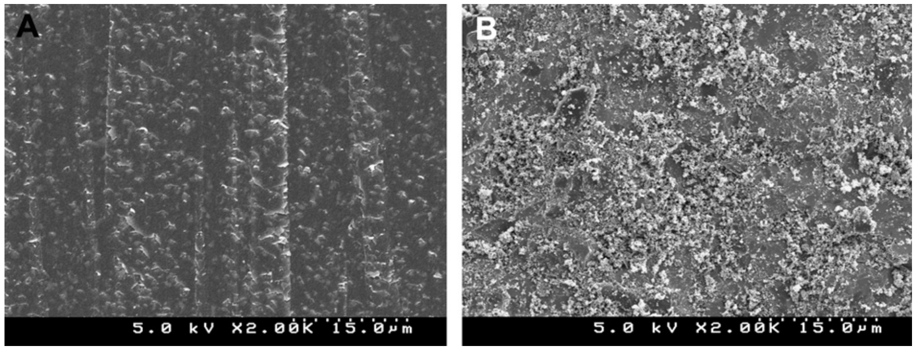

Figure 3 (A) SEM micrograph of polished zirconia surface with 600-grit polishing paper. (B) SEM micrograph of modified zirconia surface after tribochemical treatment presenting abundant particles and microporosities.

influenced by the specimen geometry and the occurrence of nonuniform stress distributions during load application.[26] Shear test measurements were used in many studies in dentistry.[28–30]

In practice, all ceramic restorations are cemented onto prepared tooth surfaces (dentin), resin composites, or metal cones. Unlike other studies utilizing a model of the zirconia-resin composite interface or zirconia-dentin interface, this study tested the bond strength between two zirconia blocks. Generally, the bond strength of cement-dentin is $<$50 MPa.[11,28] In this study, the bond strength of cement-zirconia was about 30 MPa to 70 MPa. The purpose of this study was to evaluate the bond strength to zirconia. If a zirconia-dentin tested model had been used, the result would probably have been the bond strength of cement-dentin. In order to ensure that the result was the bond strength to zirconia, this study tested the bond strength between two zirconia blocks.

With PF and FL, the degree of fatigue did not differ significantly between no cyclic impacts and compressive

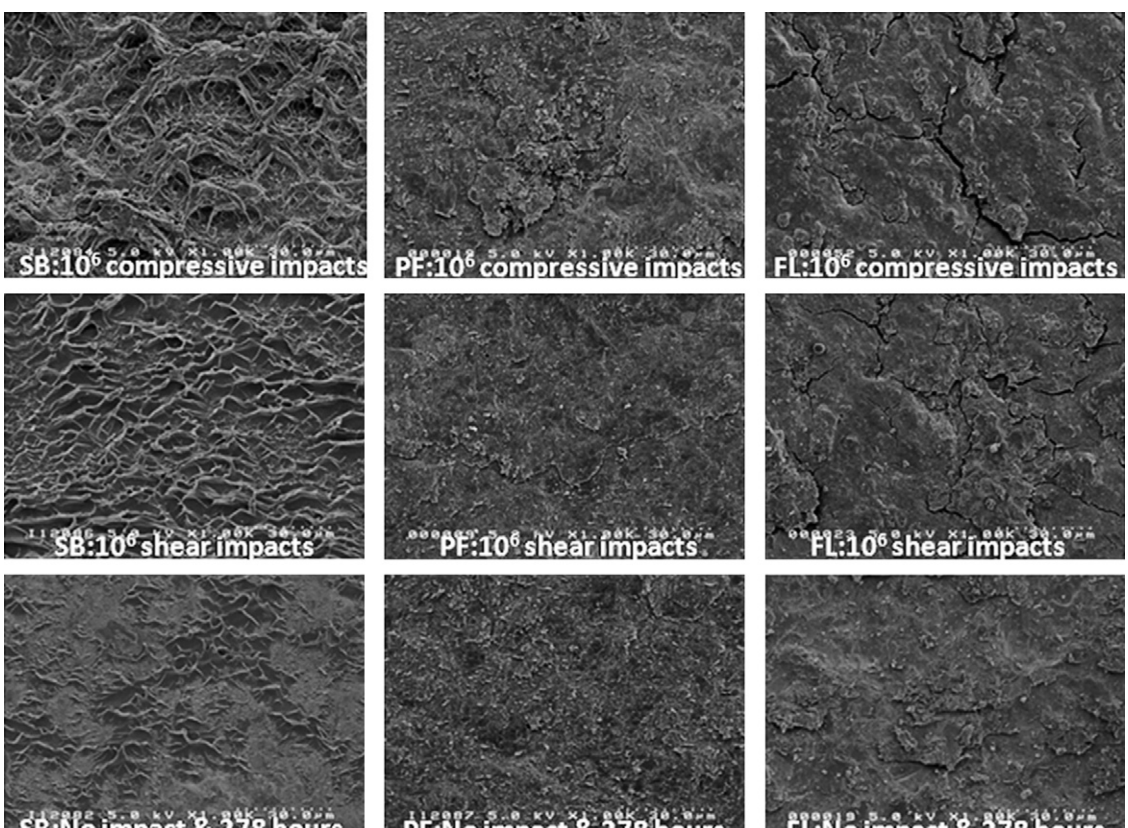

Figure 4 SEM image of the failure surface after cyclic impact. SB exhibited cup-like microcavities generally called dimples. PF showed brittle fractures of the flat surface. When no cyclic impact was applied, FL showed a flat, rough surfaces, PF and FL showed irregular cracks after CL and SL conditions.

cyclic impacts, but significantly differed between no cyclic impacts and shear cyclic impacts. Evaluation of the reduction ratio of bond strength of no cyclic impacts and shear cyclic impacts fewer than 10^6 cyclic loadings showed that the ratio varied with the adhesive resin cement tested (15% for SB, 28% for PF, and 11% for FL). With SB and FL, shear bond strengths did not significantly differ between shear cyclic impacts and compressive cyclic impacts, but significantly differed between with and without cyclic impacts. In this study, two zirconia blocks were bonded to each other, because the use of Y-TZP ceramic blocks as specimens was easier to standardize than natural teeth. Although the stress distribution pattern using dentin is not in accordance with the clinical situation, it is advisable not to apply impact loading, especially from the shear direction, until full polymerization and a high bond strength are achieved.

Qeblawi et al[16] evaluated the effects of mechanical and chemical surface treatments of zirconia on its bond strength to a resin cement and found that the highest shear bond strength values were achieved in the tribochemical treatment group. There was no significant difference between before and after 6000 thermal cycles (5–55 °C). Mirmohammadi et al[15] evaluated the effects of cyclic loading on the bond strength of resin cement to zirconia, the bonding surface of which was airborne-particle abraded. Their results revealed a 50% decrease for Panavia F 2.0 (Kuraray Medical, Tokyo, Japan) from 44 MPa to 22 MPa. This study also found that the zirconia resin bond strength is liable to deterioration under the influence of fatigue.

In this study, there was no significant difference between CL and SL with SB and FL; however, the bond strength of PF to zirconia differed statistically between CL and SL. It was reported that the elastic moduli of Panavia F2.0, which has a similar composition to PF, FL, and SB, are approximately 7.0,[31] 4.0,[31] and 1.7 GPa,[32] respectively. It seems that brittle BPEDMA resin material, PF, was more sensitive to shear cyclic impacts than compressive cyclic impacts. Results indicated that bond strengths of the resin-modified glass ionomer cement, FL, were significantly lower than those of PF and SB. The results may have been caused by the mechanical properties of resin-modified glass ionomers. It was reported that compressive and flexural strengths of resin-modifies glass ionomer are considerably lower than those of resin cement.[33]

The safety aspect is related to the asymptotic behavior of materials, many of which display a fatigue limit or endurance limit at a high number of cycles (typically $>10^6$) under benign environmental conditions.[34] The S-N curve is generally limited to 10^6 cycles, and it is admitted, according to the standard, that a horizontal asymptote allows one to determine a fatigue limit value for an alternating stress of 10^6–10^7 cycles.[34] Beyond 10^6 cycles, the standard considers that the fatigue life is infinite. In this study, although shear bond strengths subjected to 10^6 cyclic loading decreased, all luting cements tested in this study could reliably survive more than 10^6 shear or compressive cyclic impact loadings at 10 kg.

SB is an elastic material that features high viscoelasticity and high fracture toughness. SB-SL and SB-CL showed the formation of larger, more-extended dimples. The reasons for dimple formation may be that the unpolymerized monomer allowed adhesive resin cement to be extended. PF and FL are brittle materials that feature high elastic moduli and low elastic limits. PF and FL showed irregular cracks after CL and SL conditions.

Conclusions

Within the limitations of this study, the following conclusions were drawn.

1) After 10^6 compressive cyclic impact loads, MDP containing a luting system (Panavia Fluoro Cement) in combination with tribochemical treatment of zirconia surfaces produced a higher bond strength than Super Bond C&B and Fuji Luting.
2) There was no significant difference in bond strengths between Panavia Fluoro Cement and Super Bond C&B after 10^6 shear cycles.
3) After applying tribochemical treatment to zirconia, all cements used in this study survived more than 10^6 shear or compressive cyclic impact loadings of 10 kg.

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